

DEPARTMENT OF COMMERCE

BUREAU OF STANDARDS

George K. Burgess, Director

STODDARD SOLVENT

(Dry Cleaning)

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COMMERCIAL STANDARD CS3-28



ELIMINATION OF WASTE  
Through  
SIMPLIFIED COMMERCIAL PRACTICE

UNITED STATES  
GOVERNMENT PRINTING OFFICE  
WASHINGTON

1929

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Lists of the publications in each of the above series can be obtained by applying to the Commercial Standards Group, Room 316, United States Department of Commerce, Washington, D. C.

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## COMMERCIAL STANDARD CS3-28

### ACCEPTED BY

#### ASSOCIATIONS

National Association of Dyers and Cleaners.

#### INDIVIDUALS

Anderson-Prichard Oil Corporation, Oklahoma City, Okla.

Ashland Refining Co., Ashland, Ky.

Associated Oil Co., San Francisco, Calif.

Associated Refining Co. (Inc.), New York, N. Y.

Atlantic Cleaners and Dyers (Inc.), Washington, D. C.

Bartles-Maguire Oil Co., Milwaukee, Wis.

Bayburn Cleansing Shop (Inc.), Arlington, Mass.

Black Co., William E., Chicago, Ill.

Broadway Dyers and Cleaners, Portland, Oreg.

Brown & Thorn Dry Cleaning Corporation, Syracuse, N. Y.

"Browns," Cynthia, Ky.

Buck's Odorless Cleaners, Etowah, Tenn.

Bureau of Oil Inspection, St. Paul, Minn.

Butler Manufacturing Co. (Glover Sales Division), Kansas City, Mo.

Canfield Oil Co., Cleveland, Ohio.

Carnegie Refining Co., Carnegie, Pa.

Central Commercial Co. (Keystone Oil & Manufacturing Division), Chicago, Ill.

Champlin Refining Co., Enid, Okla.

Chemical Solvents (Inc.), New York, N. Y.

Clark Chemical Co., Bay City, Mich.

Clark's Laundry & Dry Cleaning Co., Mishawaka, Ind.

Cleaners Equipment Corporation, Kansas City, Mo.

Derby Oil and Refining Corporation, Wichita, Kans.

Dresher Bros., Omaha, Nebr.

Empire Laundry & Dry Cleaning Co., Augusta, Ga.

Enke's City Dye Works, Portland, Oreg.

Evans Dyeing and Cleaning Co., Brownwood, Tex.

Excelsior Laundry, Albuquerque, N. Mex.

Fink Cleaners, Lodi, Calif.

Finkelstein, Harrisburg, Pa.

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Loebl Dye Works (Inc.), Roanoke, Va.

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Mellon Institute, Pittsburgh, Pa.

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Noble Oil & Gas Co., Tulsa, Okla.

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Peterson Co., George C., Chicago, Ill.

Producers & Refiners Corporation, Independence, Kans.

Queen Cleaners & Dyers (Inc.), Detroit, Mich.  
 Rayl Co., The, Detroit, Mich.  
 Refinery Supply Co., Tulsa, Okla.  
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 Rio Grande Oil Co., Los Angeles, Calif.  
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 Schaffer Cleaning Works, Winona, Minn.  
 Schock Independent Oil Co., Mount Joy, Pa.  
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 Short Bros., Greenwood, Miss.  
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 Smith Bros., Colorado Springs, Colo.  
 Standard Oil Co. (Indiana), Chicago, Ill.  
 Standard Oil Co. of California, San Francisco, Calif.  
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Stoll Oil Refining Co., Louisville, Ky.  
 Sun Oil Co., Jacksonville, Fla.  
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 Tarr & McComb Corporation, Los Angeles, Calif.  
 Thrasher's, Mount Pleasant, Tex.  
 Union Oil Co. of California, Los Angeles, Calif.  
 United States Oil Works, Omaha, Nebr.  
 Utah Soap Co., Salt Lake City, Utah.  
 Van Winckle, James, Pontiac, Ill.  
 Wadhams Oil Co., Milwaukee, Wis.  
 Weems Laundry, Quincy, Ill.  
 Wellsville Laundry & Dry Cleaning Co., Wellsville, N. Y.  
 White Eagle Oil Refining Co., Kansas City, Mo.  
 Wichita Cleaning & Dye Works, Wichita Falls, Tex.

## GOVERNMENT

National Home for Disabled Volunteer Soldiers, Dayton, Ohio.  
 United States Department of Labor, Washington, D. C.  
 United States Veterans' Bureau, Washington, D. C.  
 War Department, Washington, D. C.



# STODDARD SOLVENT

(Dry Cleaning)

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## COMMERCIAL STANDARD CS3-28

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On December 2, 1927, a joint conference of representative manufacturers, distributors, and users adopted a commercial standard for Stoddard solvent. The industry has since accepted and approved for promulgation by the Department of Commerce the specification as shown herein.

### I. MATERIAL

Stoddard solvent shall be a petroleum distillate conforming to the detailed requirements given herein.

### II. DETAILED REQUIREMENTS

Stoddard solvent as received shall conform to the following requirements when tested according to the methods stated in Section III of this specification:

#### 1. APPEARANCE

Shall be clear and free from suspended matter and undissolved water.

#### 2. COLOR

Shall be water-white or not darker than 21 by Saybolt chromometer.

#### 3. ODOR

Shall be sweet.

#### 4. FLASH POINT

Shall be not lower than 100° F. ("Tag" closed tester.)

#### 5. CORROSION TEST

A clean copper strip shall show not more than extremely slight discoloration when submerged in the solvent for three hours at 212° F.

#### 6. DISTILLATION RANGE

Not less than 50 per cent shall be recovered in the receiver when the thermometer reads 350° F. The dry or end point shall be not higher than 410° F. No tolerance shall be allowed above 410° F.

#### 7. ACIDITY

The residue remaining in the flask after the distillation is completed shall not show an acid reaction.

## 8. DOCTOR TEST

Shall be negative.

## 9. SULPHURIC ACID ABSORPTION TEST

Not more than 5 per cent of the solvent shall be absorbed by concentrated "c. p." sulphuric acid (specific gravity, 1.835) (approximately 93.2 per cent).

III. METHODS OF INSPECTION AND TESTING<sup>1</sup>

## 1. DETENTION AND REMOVAL OF SEPARATED WATER

Draw a portion of the solvent by means of a glass or metal container with a removable stopper or top, or with a "thief," from the lowest part of the container, or by opening the bottom valve of the perfectly level tank car. If water is found to be present, draw it all out, record the quantity, and deduct it from the total volume of liquid delivered.

## 2. SAMPLING

The method of sampling given under (a) should be used whenever feasible. When method (a) is not applicable, method (b), (c), or (d) is to be used, according to the special conditions that obtain.

(a) WHILE LOADING TANK CAR OR WHILE FILLING CONTAINERS FOR SHIPMENT.—Samples shall be drawn by the purchaser's inspector at the discharge pipe where it enters the receiving vessel or vessels. The composite sample shall be not less than 5 gallons and shall consist of small portions of not more than 1 quart each taken at regular intervals during the entire period of loading or filling. The composite sample thus obtained shall be thoroughly mixed, and from it three samples of not less than 1 quart each shall be placed in clean, dry, glass bottles or tin cans, which must be nearly filled with the sample and securely stoppered with new, clean corks or well-fitting covers or caps. These shall be sealed and distinctly labeled by the inspector; one shall be delivered to the buyer, one to the seller, and the third held for check in case of dispute.

(b) FROM LOADED TANK CAR OR OTHER LARGE VESSEL.—A composite sample of not less than 5 gallons shall be made up of numerous small samples of not more than 1 quart each taken from the top, bottom, and intermediate points by means of a metal or glass container with removable stopper or top. This device, attached to a suitable pole, is lowered to the various desired depths, when the stopper or top is removed and the container allowed to fill. The sample thus obtained is handled as in (a).

<sup>1</sup> The methods given are from the standards of the American Society for Testing Materials and from Federal Specifications Board specifications Nos. 2d and 7 (Bureau of Mines Technical Paper 323B and Bureau of Standards Circular No. 86).



(c) **BARRELS AND DRUMS.**—Barrels and drums shall be sampled after gauging contents. Five per cent of the packages in any shipment or delivery shall be represented in the sample. Thoroughly mix the contents of each barrel to be sampled by stirring with a clean rod and withdraw a portion from the center by means of a "thief" or other sampling device. The composite sample thus obtained shall be not less than 3 quarts, shall consist of equal portions of not less than one-half pint from each package sampled, and shall be handled as in (a). Should the inspector suspect adulteration, he shall draw the samples from the suspected packages.

(d) **SMALL CONTAINERS, CANS, ETC., OF 10 GALLONS OR LESS.**—These should be sampled, while filling, by method (a) whenever possible, but in case this is impossible the composite sample taken shall be not less than 3 quarts. This shall be drawn from at least five packages (from all when fewer), and in no case from less than 2 per cent of the packages. The composite sample thus taken shall be thoroughly mixed and subdivided as in (a).

### 3. LABORATORY EXAMINATION

(a) **APPEARANCE.**—Examine to determine compliance with the specifications.

(b) **COLOR.**—Color shall be determined by the Saybolt chromometer A. S. T. M. method D156-23T. (Twenty-one Saybolt color is the equivalent of a freshly prepared solution of potassium bichromate ( $K_2Cr_2O_7$ ) in distilled water containing 0.0048 grams per liter.)

(c) **ODOR.**—Note whether the sample conforms to the specification.

(d) **FLASH POINT BY THE TAG CLOSED TESTER** (A. S. T. M. METHOD D56-21).—(Bureau of Mines Technical Paper 323B, method 110.11.)

#### APPARATUS

1. (a) The tag closed tester (see fig. 12, Bureau of Mines Technical Paper 323B) shall conform to the following dimensions within the limits of tolerances given:

	Normal	Tolerances
	<i>Inches</i>	<i>Inches</i>
Depth of water surface below top of cup.....	$\left\{ \begin{array}{l} 1\frac{3}{32} \\ (27.8 \text{ mm}) \end{array} \right.$	$\left\{ \begin{array}{l} \pm\frac{1}{64} \\ (0.4 \text{ mm}) \end{array} \right.$
Depth of oil surface below top of cup <sup>1</sup> .....	$\left\{ \begin{array}{l} 1\frac{3}{32} \\ (29.4 \text{ mm}) \end{array} \right.$	$\left\{ \begin{array}{l} \pm\frac{1}{64} \\ (0.4 \text{ mm}) \end{array} \right.$
Depth of top of bulb of oil thermometer when in place below top of cup.....	$\left\{ \begin{array}{l} 1\frac{1}{16} \\ (33.3 \text{ mm}) \end{array} \right.$	$\left\{ \begin{array}{l} \pm\frac{1}{32} \\ (0.8 \text{ mm}) \end{array} \right.$
Inside diameter of oil cup at top.....	$\left\{ \begin{array}{l} 2\frac{1}{8} \\ (54 \text{ mm}) \end{array} \right.$	$\left\{ \begin{array}{l} \pm 0.005 \\ (0.1 \text{ mm}) \end{array} \right.$
Diameter of bead on top of cover.....	$\left\{ \begin{array}{l} \frac{9}{32} \\ (4 \text{ mm}) \end{array} \right.$	$\left\{ \begin{array}{l} \pm\frac{1}{64} \\ (0.4 \text{ mm}) \end{array} \right.$
Weight of oil cup.....	$\left\{ \begin{array}{l} \text{Grams} \\ 68 \end{array} \right.$	$\left\{ \begin{array}{l} \text{Grams} \\ \pm 1 \end{array} \right.$

<sup>1</sup> This is the phraseology of the method as published. "Oil" is taken to mean gasoline, naphtha, or any other petroleum distillate. This comment holds throughout this specification whenever the description of a method is quoted.

The plane of underside of cover to be between the top and bottom of the burner tip when the latter is fully depressed.

(b) The thermometer shall conform to the following specifications: These specifications cover a special thermometer graduated in Fahrenheit degrees, the range being  $+20$  to  $+230^{\circ}$  F.

*Type*.—Etched stem, glass.

*Liquid*.—Mercury.

*Range and subdivision*.— $+20$  to  $+230^{\circ}$  F. in  $1^{\circ}$  F.

*Total length*.—273 to 277 mm (10.75 to 10.92 inches).

*Stem*.—Plain front, enamel back, suitable thermometer tubing; diameter, 6 to 7 mm (0.24 to 0.28 inch).

*Bulb*.—Corning normal or equally suitable thermometric glass; length 9 to 13 mm (0.35 to 0.51 inch); diameter, not greater than stem.

Distance to  $+20^{\circ}$  F. line from bottom of bulb, 75 to 90 mm (2.93 to 3.54 inches).

Distance to  $230^{\circ}$  F. line from top of thermometer, 25 to 40 mm (0.98 to 1.57 inches).

*Expansion chamber*.—To permit heating the thermometer at least  $90^{\circ}$  F. above highest temperature on scale.

*Filling above mercury*.—Nitrogen gas.

*Top finish*.—Glass ring.

*Graduation*.—All lines, figures, and letters clear cut and distinct; the first and each succeeding  $5^{\circ}$  F. line to be longer than the remaining lines, graduations to be numbered at each multiple of  $10^{\circ}$  F.

*Immersion*.—57 mm or  $2\frac{1}{4}$  inches. The words " $2\frac{1}{4}$ -inch immersion" on Fahrenheit thermometers and a line around the stem 57 mm or  $2\frac{1}{4}$  inches above the bottom of the bulb shall be etched on the thermometer.

*Special marking*<sup>2</sup>.—"A. S. T. M., P. M., and Tag," a serial number and the manufacturer's name or trade-mark shall be etched on the stem.

*Scale error*.—The error at any point of the scale when the thermometer is standardized as provided below shall not exceed  $1^{\circ}$  F.

*Standardization*.—The thermometer shall be standardized at the ice point and at intervals of approximately  $50^{\circ}$  F. for  $2\frac{1}{4}$ -inch immersion and for the following temperatures of the emergent mercury column.

Thermometer reading	Average temperature of emergent mercury column
$^{\circ}$ F.	$^{\circ}$ F.
70	70
100	86
150	104
212	118

<sup>2</sup> This thermometer is identical with the low-range Pensky-Martens instrument described in A. S. T. M. method D93-22.

*Case.*—The thermometer shall be supplied in a suitable case on which shall appear the marking “A. S. T. M., P. M., and Tag, 20° to 230° F.”

*NOTE.*—For the purpose of interpreting these specifications the following definitions apply: The total length is the over-all length of the finished instrument. The diameter is that measured with a ring gauge. The length of the bulb is the distance from the bottom of the bulb to the beginning of the enamel backing. The top of the thermometer is the top of the finished instrument.

#### PROCEDURE

2. (a) If gas is available, connect a  $\frac{1}{8}$ -inch rubber tube to the corrugated gas connection on the oil-cup cover. If no gas is available, unscrew the test-flame burner tip from the oil chamber on the cover and insert a wick of cotton cord in the burner tip and replace it. Put a small quantity of cotton waste in the oil chamber, insert a small quantity of signal, sperm, or lard oil in the chamber, light the wick, and adjust the flame so that it is exactly the size of the small white bead mounted on the top of the tester.

(b) The test shall be performed in a dim light, so that the flash may be seen plainly.

(c) Surround the tester on three sides with an inclosure to keep away drafts. A shield about 18 inches square and 2 feet high, open in front, is satisfactory, but any safe precaution against all possible room drafts is acceptable. Tests made in a laboratory hood or near ventilators will give unreliable results.

(d) See that the tester sits firm and level.

(e) For accuracy, the flash-point thermometers which are especially designed for the instrument should be used, as the position of the bulb of the thermometer in the oil cup is essential.

3. Put the water-bath thermometer in place and put a receptacle under the overflow spout to catch the overflow. Fill the water bath with water at such a temperature that, when testing is started, the temperature of the water bath will be at least 20° F. (11° C.) below the probable flash point of the oil to be tested.

4. Put the oil cup in place in the water bath. Measure 50 milliliters (ml)<sup>3</sup> of the oil to be tested in a pipette or a graduate and place in the oil cup. The temperature of the oil shall be at least 20° F. (11° C.) below its probable flash point when testing is started. Destroy any bubbles on the surface of the oil. Put on the cover, with flash-point thermometer in place and gas tube attached. Light the pilot light on the cover and adjust the flame to the size of the small white bead on the cover.

<sup>3</sup> In all volumetric work the unit of volume employed is the milliliter (ml), the one-thousandth part of the liter. The liter is defined as the volume occupied by a quantity of pure water at 4° C. having a mass of 1 kilogram. Milliliter is often incorrectly called cubic centimeter (cc). The designation “ml” will be used throughout this specification.



5. Light and place the heating lamp, filled with alcohol, in the base of the tester and see that it is centrally located. Adjust the flame of the alcohol lamp so that the temperature of the oil in the cup rises at the rate of about  $1.8^{\circ}\text{ F.}$  ( $1^{\circ}\text{ C.}$ ) per minute, not faster than  $2^{\circ}\text{ F.}$  ( $1.1^{\circ}\text{ C.}$ ), nor slower than  $1.6^{\circ}\text{ F.}$  ( $0.9^{\circ}\text{ C.}$ ) per minute.

6. (a) Record the barometric pressure, which, in the absence of a laboratory instrument, may be obtained from the nearest Weather Bureau station.

(b) Record the temperature of the oil sample at start.

(c) When the temperature of the oil reaches  $9^{\circ}\text{ F.}$  ( $5^{\circ}\text{ C.}$ ) below the probable flash point of the oil, turn the knob on the cover so as to introduce the test flame into the cup and turn it promptly back again. Do not let it snap back. The time consumed in turning the knob down and back should be about one full second, or the time required to pronounce distinctly the words "one-thousand-and-one."

(d) Record the time of making the first introduction of the test flame.

(e) Record the temperature of the oil sample at the time of first test.

(f) Repeat the application of the test flame at every  $1^{\circ}\text{ F.}$  ( $0.5^{\circ}\text{ C.}$ ) rise in temperature of the oil until there is a flash of the oil within the cup. Do not be misled by an enlargement of the test flame or a halo around it when it enters the cup, or by slight flickering of the flame; the true flash consumes the gas in the top of the cup and causes a very slight explosion.

(g) Record the time at which the flash point is reached.

(h) Record the flash point.

(i) If the rise in temperature of the oil from the "time of making the first introduction of the test flame" to the "time at which the flash point is reached" was faster than  $2^{\circ}\text{ F.}$  ( $1.1^{\circ}\text{ C.}$ ) or slower than  $1.6^{\circ}\text{ F.}$  ( $0.9^{\circ}\text{ C.}$ ) per minute, the test should be questioned and the alcohol heating lamp adjusted to correct the rate of heating. It will be found that the wick of this lamp can be so accurately adjusted as to give a uniform rate of rise in temperature within the above limits and remain so.

7. (a) It is not necessary to turn off the test flame with the small regulating valve on the cover; leave it adjusted to give the proper size of flame.

(b) Having completed the preliminary test, remove the heating lamp, lift up the oil-cup cover, and wipe off the thermometer bulb. Lift out the oil cup and empty and carefully wipe it. Throw away all oil samples after they have been once used in making a test.

(c) Pour cold water into the water bath, allowing it to overflow into a receptacle until the temperature of the water in the bath is lowered  $15^{\circ}\text{ F.}$  ( $8^{\circ}\text{ C.}$ ) below the flash point of the oil, as shown

by the previous test. With cold water of nearly constant temperature, it will be found that a uniform amount will be required to reduce the temperature of the water bath to the required point.

(d) Place the oil cup back in the bath and measure into it a 50 ml charge of fresh oil. Destroy any bubbles on the surface of the oil, put on the cover with its thermometer, put in the heating lamp, record the temperature of the oil, and proceed to repeat the test as described above in sections 4 to 6, inclusive. Introduce the test flame for the first time at a temperature of 10° F. (5.5° C.) below the flash point obtained on the previous test.

8. If two or more determinations agree within 1° F. (0.5° C.), the average of these results, corrected for barometric pressure, shall be considered the flash point. If two determinations do not check within 1° F. (0.5° C.), a third determination shall be made, and if the maximum variation of the three tests is not greater than 2° F. (1° C.), their average, after correcting for barometric pressure, shall be considered the flash point.

Correction for barometric pressure shall be made only in cases of dispute or when the barometer reading varies more than one-half inch (13 mm) from the standard pressure of 29.92 inches (760 mm). When the barometer reading is below this standard pressure add to the thermometer reading 1.6° F. (0.9° C.) for each inch (25 mm) of barometer difference to obtain the true flash point. When the barometer reading is above the standard pressure deduct 1.6° F. (0.9° C.) for each inch (25 mm) of barometer difference to obtain the true flash point.

(e) CORROSION TEST AT 212° F.<sup>4</sup> (COPPER STRIP).—Place a clean strip of mechanically polished pure sheet copper about ½ inch wide and 3 inches long in a clean test tube. Add enough of the sample to be tested completely to cover the strip. Close the tube with a vented stopper and hold in a thermostat for 3 hours at 212° F. Rinse the copper strip with sulphur-free acetone and compare it with similar strip of freshly polished copper. Discoloration or pitting indicates corrosion.

(f) DISTILLATION<sup>5</sup> (APPARATUS).—(1) *Flask*.—The standard 100 ml Engler flask is shown in Figure 10, Bureau of Mines Technical Paper 323B, the dimensions and allowable tolerance being as follows:

*Dimensions of Engler flask*

Description	Centi- meters	Inches	Toler- ances
			<i>Cm</i>
Diameter of bulb, outside.....	6.5	2.56	0.2
Diameter of neck, inside.....	1.6	.63	.1
Length of neck.....	15.0	5.91	.4
Length of vapor tube.....	10.0	3.94	.3
Diameter of vapor tube, outside.....	.6	.24	.05
Diameter of vapor tube, inside.....	.4	.16	.05
Thickness of vapor tube-wall.....	.1	.04	.05

<sup>4</sup> Method 530.31 Bureau of Mines Technical Paper 323B.

<sup>5</sup> Method 100.13 Bureau of Mines Technical Paper 323B. (A. S. T. M. method 186-27.)



The position of the vapor tube shall be 9 cm (3.55 inches)  $\pm$  3 mm above the surface of the liquid when the flask contains its charge of 100 ml. The tube is approximately in the middle of the neck and set at an angle of  $75^\circ$  (tolerance  $\pm 3^\circ$ ) with the vertical.

(2) *Condenser*.—The condenser (see fig. 11 Bureau of Mines Technical Paper 323B) consists of a  $\frac{1}{8}$ -inch (14.29 mm) outside diameter No. 20 Stubbs gauge seamless brass tube 22 inches (55.88 cm) long. It is set at an angle of  $75^\circ$  from the perpendicular and is surrounded with a cooling bath 15 inches (38.1 cm) long, approximately 4 inches (10.16 cm) wide by 6 inches (15.24 cm) high. The lower end of the condenser tube is cut off at an acute angle, and curved downward for a length of 3 inches (7.62 cm) and slightly backward so as to insure contact with the wall of the graduate at a point 1 to  $1\frac{1}{4}$  inches (2.54 to 3.8 cm) below the top of the graduate when it is in position to receive the distillate.

(3) *Shield*.—The shield (see fig. 11 Bureau of Mines Technical Paper 323B) is made of approximately 22-gauge sheet metal and is 19 inches (48.26 cm) high, 11 inches (27.94 cm) long, and 8 inches (20.32 cm) wide, with a door on one narrow side, with two openings; 1 inch (2.54 cm) in diameter, equally spaced in each of the two narrow sides, and with a slot cut in one side for the vapor tube. The centers of these four openings are  $8\frac{1}{2}$  inches (21.59 cm) below the top of the shield. There are also three  $\frac{1}{2}$ -inch (1.27 cm) holes in each of the four sides with their centers 1 inch (2.54 cm) above the base of the shield.

(4) *Ring support and hard asbestos boards*.—The ring support is of the ordinary laboratory type, 4 inches (10.16 cm) or larger in diameter, and is supported on a stand inside the shield. There are two hard asbestos boards; one 6 by 6 by  $\frac{1}{4}$  inch (15.24 by 15.24 cm by 6.35 mm), with a hole  $1\frac{1}{4}$  inches (3.175 cm) in diameter in its center, the sides of which shall be perpendicular to the surface; the other, an asbestos board to fit tightly inside the shield, with an opening 4 inches (10.16 cm) in diameter concentric with the ring support. These are arranged as follows: The second asbestos board is placed on the ring and the first or smaller asbestos board on top, so that it may be moved in accordance with the directions for placing the distilling flask. Direct heat is applied to the flask only through the  $1\frac{1}{4}$ -inch (3.175 cm.) opening in the first asbestos board.

(5) *Gas burner or electric heater*.—(a) *Gas burner*.—The burner is so constructed that sufficient heat can be obtained to distill the product at the uniform rate specified below. The flame should never be so large that it spreads over a circle of diameter greater than  $3\frac{1}{2}$  inches (8.89 cm) on the under surface of the asbestos board. A sensitive regulating valve is a necessary adjunct, as it gives complete control of heating.

(b) *Electric heater.*—The electric heater, which may be used in place of the gas flame, shall be capable of bringing over the first drop within the time specified below when started cold and of continuing the distillation at the uniform rate. The electric heater shall be fitted with an asbestos board top  $\frac{1}{8}$  to  $\frac{1}{4}$  inch (3.175 to 6.35 mm) thick, having a hole  $1\frac{1}{4}$  inches (3.175 cm) in diameter in the center. When an electric heater is employed, the portion of the shield above the asbestos board shall be the same as with the gas burner, but the part below the board may be omitted.

(6) *Thermometer.*—(a) The A. S. T. M. low-distillation thermometer shall conform to the following requirements: These specifications cover a total immersion thermometer graduated in either centigrade or Fahrenheit degrees, as specified, the ranges being 0 to 300° C. or 30 to 580° F., respectively.

*Type.*—Etched stem, glass.

*Liquid.*—Mercury.

*Range and subdivision.*—0 to 300° C. in 1° C., or 30 to 580° F. in 2° F.

*Total length.*—378 to 384 mm (14.88 to 15.12 inches).

*Stem.*—Plain front, enamel back, suitable thermometer tubing; diameter, 6 to 7 mm (0.24 to 0.28 inch).

*Bulb.*—Corning normal or equally suitable thermometric glass; length, 10 to 15 mm (0.39 to 0.59 inch); diameter, 5 to 6 mm (0.20 to 0.24 inch).

*Distance to 0° C. or 32° F. line from bottom of bulb.*—100 to 110 mm (3.94 to 4.33 inches).

*Distance to 300° C. or 572° F. line from top of thermometer.*—30 to 45 mm (1.18 to 1.77 inches).

*Filling above mercury.*—Nitrogen gas.

*Top finish.*—Glass ring.

*Graduation.*—All lines, figures, and letters clear cut and distinct; the first and each succeeding 5° C. or 10° F. line to be longer than the remaining lines; graduations to be numbered at each multiple of 10° C. or 20° F.

*Immersion.*—Total.

*Special marking.*—“A. S. T. M. low distillation,” a serial number, and the manufacturer’s name or trade-mark shall be etched on the stem.

*Scale error.*—The error at any point of the scale when the thermometer is standardized as provided below shall not exceed 0.5° C. or 1° F.

*Standardization.*—The thermometer shall be standardized immersed in the testing bath to the top of the mercury column, at the ice point and at temperature intervals of approximately 50° C. or 100° F. up to 300° C. or 572° F.

*Test for permanence of range.*—After being subjected to a temperature of 280 to 290° C. or 540 to 560° F. for 24 hours the accuracy shall be within the limit specified.

*Case.*—The thermometer shall be supplied in a suitable case on which shall appear the marking "A. S. T. M. low distillation, 0 to 300° C." or "A. S. T. M. low distillation, 30 to 580° F.," according to the type of thermometer.

*NOTE.*—For the purpose of interpreting these specifications the following definitions apply: The total length is the over-all length of the finished instrument. The diameter is that measured with a ring gauge. The length of the bulb is the distance from the bottom of the bulb to the beginning of the enamel backing. The top of the thermometer is the top of the finished instrument.

(7) *Graduate.*—The graduate shall be of the cylindrical type, of uniform diameter, with a pressed or molded base and a lipped top. The cylinder shall be graduated to contain 100 ml, and the graduated portion shall be not less than 7 inches (17.78 cm) nor more than 8 inches (20.32 cm) long; it shall be graduated in single milliliters, and each fifth mark shall be distinguished by a longer line. It shall be numbered from the bottom up at intervals of 10 ml. The over-all height of the graduate shall be not less than 9¼ inches (24.8 cm) nor more than 10¼ inches (26.0 cm). The graduations shall not be in error by more than 1 ml at any point on the scale.

#### PROCEDURE

(8) (a) The condenser bath shall be filled with cracked ice and enough water added to cover the condenser tube. The temperature shall be maintained between 32 and 40° F. (0 and 4.45° C.).

(b) The condenser tube shall be swabbed to remove any liquid remaining from the previous test. A piece of soft cloth attached to a cord or copper wire may be used for this purpose.

(c) One hundred milliliters of the product shall be measured in the 100 ml graduated cylinder at 55 to 65° F. (12.78 to 18.33° C.) and transferred directly to the Engler flask. None of the liquid shall be permitted to flow into the vapor tube.

(d) The thermometer provided with a cork shall be fitted tightly into the flask, so that it will be in the middle of the neck and so that the lower end of the capillary tube is on a level with the inside of the bottom of the vapor outlet tube at its junction with the neck of the flask. The thermometer shall be approximately at room temperature when placed in the flask.

(e) The charged flask shall be placed in the 1¼-inch (3.175 cm) opening in the 6 by 6 inch (15.24 by 15.24 cm) asbestos board with the vapor outlet tube inserted into the condenser tube. A tight connection may be made by means of a cork through which the vapor tube passes. The position of the flask shall be so adjusted that the



vapor tube extends into the condenser tube not less than 1 inch (2.54 cm) nor more than 2 inches (5.08 cm).

(f) The graduated cylinder used in measuring the charge shall be placed, without drying, at the outlet of the condenser tube in such a position that the condenser tube shall extend into the graduate at least 1 inch (2.54 cm), but not below the 100 ml mark. Unless the temperature is between 55 and 65° F. (12.78 and 18.33° C.) the receiving graduate shall be immersed up to the 100 ml mark in a transparent bath maintained between these temperatures. The top of the graduate shall be covered closely during the distillation with a piece of blotting paper or its equivalent, cut so as to fit the condenser tube tightly.

(9) When everything is in readiness, heat shall be applied at a uniform rate, so regulated that the first drop of condensate falls from the condenser in not less than 5 nor more than 10 minutes. When the first drop falls from the end of the condenser, the reading of the distillation thermometer shall be recorded as the initial boiling point. The receiving cylinder shall then be moved so that the end of the condenser tube shall touch the side of the cylinder. The heat shall then be so regulated that the distillation will proceed at a uniform rate of not less than 4 nor more than 5 ml per minute. The volume of distillate collected in the cylinder shall be observed and recorded to the nearest 0.5 ml when the mercury of the thermometer reaches each 10 ml mark on the graduate.

After the 90 per cent point has been recorded the heat may be increased because of the presence of the heavy ends which have relatively high boiling points. However, no further increase of heat shall be applied after this adjustment. The 4 to 5 ml rate can rarely be maintained from the 90 per cent point to the end of the distillation, but in no case shall the period between the 90 per cent point and the end point be more than 5 minutes.

The heating shall be continued until the mercury reaches a maximum and starts to fall consistently. The highest temperature observed on the distillation thermometer shall be recorded as the maximum temperature or end point. Usually this point will be reached after the bottom of the flask has become dry.

The total volume of the distillate collected in the receiving graduate shall be recorded as the recovery.

The cooled residue shall be poured from the flask into a small cylinder graduated in 0.1 ml, measured when cool, and the volume recorded as residue.

The difference between 100 ml and the sum of the recovery and the residue shall be calculated and recorded as distillation loss.

#### ACCURACY

(10) With proper care and attention to detail, duplicate results obtained for maximum temperature should not differ from each other by more than 6° F. (3.33° C.). Duplicate temperature readings

at the various percentage points should not differ by more than the amounts equivalent to 2 ml of distillate at each point in question and shall not exceed 410° F.

#### CORRECTION FOR BAROMETRIC PRESSURE

(11) The actual barometric pressure shall be ascertained and recorded, but no correction shall be made except in case of dispute. In such cases the temperature points shall be corrected to 760 mm (29.92 inches) by the use of the Sydney Young equation, as follows:

For centigrade readings:

$$C_c = 0.00012 (760 - P (273 + t_c))$$

For Fahrenheit readings:

$$C_f = 0.00012 (760 - P (460 + t_f))$$

in which  $C_c$  and  $C_f$  are, respectively, corrections to be added to the observed temperature  $t_c$  or  $t_f$ , and  $P$  is the actual barometric pressure in millimeters of mercury.

The following table is a convenient approximation of the corrections as calculated by the above equation:

Temperature range		Correction <sup>1</sup> per 10 mm difference in pressure		Temperature range		Correction <sup>1</sup> per 10 mm difference in pressure	
°C.	°F.	°C.	°F.	°C.	°F.	°C.	°F.
10-30	50-86	0.35	0.63	210-230	410-446	.59	1.06
30-50	86-122	.38	.68	230-250	446-482	.62	1.11
50-70	122-158	.40	.72	250-270	482-518	.64	1.15
70-90	158-194	.42	.76	270-290	518-554	.66	1.19
90-110	194-230	.45	.81	290-310	554-590	.69	1.24
110-130	230-266	.47	.85	310-330	590-626	.71	1.28
130-150	266-302	.50	.89	330-350	626-662	.74	1.32
150-170	302-338	.52	.94	350-370	662-698	.76	1.37
170-190	338-374	.54	.98	370-390	698-734	.78	1.41
190-210	374-410	.57	1.02	390-410	734-770	.81	1.45

<sup>1</sup> To be added if barometric pressure is below 760 mm; to be subtracted if barometric pressure is above 760 mm.

(g) ACIDITY <sup>6</sup>—Collect in a test tube the cooled residue from the distillation flask, add three volumes of distilled water, and shake the tube thoroughly. Allow the mixture to separate and remove the aqueous layer to a clean test tube by means of a pipette. Add 1 drop of a 1 per cent solution of methyl orange. No pink or red color shall be formed. The production of a pink or red color indicates the presence of a mineral acid, usually sulphuric.

NOTE.—This test is to be made immediately after distillation test.

#### PREPARATION OF REAGENTS

(h) DOCTOR TEST (SODIUM PLUMBITE).<sup>7</sup>—(1) *Sodium plumbite (doctor solution)*.—Dissolve approximately 125 g of sodium hydroxide

<sup>6</sup> Method 510.2 Bureau of Mines Technical Paper 323B.

<sup>7</sup> Method 520.3 Bureau of Mines Technical Paper 323B.





(3) One 10 ml pipette standardized to agree with stoppered Babcock bottle specified above.

#### MAKING OF TEST

Bring the temperature of the sample to 20° C. (plus or minus 1° C.). Measure out 10 ml of the sample into the clean dry modified Babcock bottle with the standard pipette and cool in ice water for five minutes. Add from a graduate 20 ml of c. p. sulphuric acid of 1.83 to 1.84 specific gravity at 20° C. (approximately 93.2 per cent). The acid should be poured down the side of the bottle to prevent splashing. Again cool by allowing the bottle to stand in ice water for 10 minutes, so that the water level is above the level of the sample in the bottle. Remove the Babcock bottle from the water bath, place glass stopper previously wet with sulphuric acid in bottle and shake it violently for one minute. Carefully add to the bottle sufficient sulphuric acid to bring the liquid level almost to the top graduations, and allow the stoppered bottle to stand over night (at least 12 hours is necessary).

Place the bottle in a water bath at 20° C. (plus or minus 1° C.) for 15 minutes. Add sulphuric acid, previously brought to the temperature of 20° C., to bring the liquid level exactly to the top graduations. Read the scale at the lower surface of the solvent and report as percentage absorbed in sulphuric acid.

#### IV. PACKING AND MARKING

Packing shall be in accordance with commercial practice unless otherwise specified.

This standard is effective from March 1, 1928, subject to regular annual revision by the standing committee.

Promulgation recommended.

R. M. HUDSON,

*Assistant Director, Commercial Standards.*

Promulgated.

GEORGE K. BURGESS,

*Director, Bureau of Standards.*

Approved February 29, 1928.

HERBERT HOOVER,

*Secretary of Commerce.*

#### HISTORY OF PROJECT

With a view to reducing the loss of life, property damage, and other fire hazards in the dry-cleaning industry, W. J. Stoddard, of Atlanta, Ga., assisted by Lloyd E. Jackson, senior industrial fellow, of the Mundatechnical Society of America, carried out intensive studies and tests of various petroleum distillates in the laboratories of the Mellon

Institute of Industrial Research, Pittsburgh, Pa., and in his dry-cleaning plant at Atlanta. As a result of this work a recommended specification for a satisfactory and comparatively safe dry-cleaning solvent was announced in May, 1925.

The National Association of Dyers and Cleaners adopted the name "Stoddard solvent" for this product in view of the personal sacrifices of time and money made by President Stoddard in developing it. The National Association of Dyers and Cleaners has supported research associates at the Bureau of Standards for several years, and through this contact the bureau has assisted indirectly, if not directly, in furthering the development and use of specifications for this solvent.

Several dry-cleaning plants having used solvent purchased under the specification, it soon became apparent that the specification should be made more definite in certain requirements, should be revised in others, and should contain detailed methods of sampling and testing, in order to avoid misinterpretation of contracts and confusion in testing laboratories. The National Association of Dyers and Cleaners called on the Bureau of Standards to assist in making these changes and additions. A tentative draft of a specification so revised was accordingly prepared in cooperation with representatives of the association. Among the individuals especially active in the formulation of the specification are the following:

W. J. Stoddard, National Association of Dyers and Cleaners.

C. C. Hubbard, National Association of Dyers and Cleaners.

I. M. Tull, National Association of Dyers and Cleaners.

Lloyd E. Jackson, Mellon Institute, Pittsburgh, Pa.

A. J. Kraemer, Bureau of Mines.

W. E. Emley, H. T. Kennedy, C. E. Waters, P. H. Walker, and F. W. Smither, of the Bureau of Standards.

## GENERAL CONFERENCE

[Washington, D. C., December 2, 1927]

In compliance with request of the National Association of Dyers and Cleaners, dated November 1, 1927, a general conference of all representative interests was held on December 2, 1927, at the Department of Commerce, Washington, D. C.

The tentative specifications transmitted as a part of the agenda for the meeting were modified as regards color of the solvent which shall be not darker than 21 by Saybolt chromometer. The initial point of 300° F. was deleted, as it was the consensus of opinion that the 100° F. flash point covers this with sufficient accuracy.

No definite agreement was reached with regard to amount of unsaturates to be permitted or the methods of test to be used in this connection. The conference, therefore, delegated this matter to the



following subcommittee, with the understanding that the conference would abide by their decision.

W. J. Stoddard, chairman, National Association of Dyers and Cleaners.

Ludlow Clayden, Sun Oil Co.

H. M. Hancock, Atlantic Refining Co.

E. E. Follin, Standard Oil Co. of Indiana.

Lloyd Jackson, Mellon Institute.

C. C. Hubbard, National Association of Dyers and Cleaners.

H. T. Kennedy, Bureau of Standards.

The above committee reported its final recommendations on March 2 to the effect that not more than 5 per cent of the solvent shall be absorbed by concentrated c. p. sulphuric acid and indicated the exact methods of test to be used which have been incorporated in the specification.

The conference indorsed the specification as revised subject to changes approved by the subcommittee and voted to have the "certification plan" put into operation on the basis of the standard.

#### STANDING COMMITTEE AND EFFECTIVE DATE

The conference then appointed the same committee listed above to act as the standing committee, this committee to hold annual meetings for the purpose of considering comments and suggested changes in the existing standard in order that it may be kept continuously in accord with the desires of the industry and advance in the art.

The conference set March 1, 1928, as the date for the specification to become effective and accepted one year from effective date as the interval for revision.

The question of developing potential foreign markets for this commodity by means of translation of the specification was considered with the result that the conference suggested that the specification be used at home for at least one year before making special effort to advertise it abroad.

Those who attended the general conference were:

AGNEW, Dr. P. G., American Engineering Standards Committee.

ANDREE, W. J., Sinclair Refining Co.

BANKER, Col. E. W., Marine Corps, Navy Department.

BARNES, THOMAS J., Sun Oil Co.

CLAYDEN, A. LUDLOW, Sun Oil Co.

DEAN, Dr. E. W., Standard Oil Co. of New Jersey.

DERBYSHIRE, L. C., Sinclair Refining Co.

FANTOZZI, C. T., Shaffer Oil & Refining Co.

FITZWILSON, C. B., Ryder Dry Cleaning Co.

FOLLIN, E. E., Standard Oil Co. of Indiana.

HANCOCK, H. M., Atlantic Refining Co.

HUBBARD, C. C., National Association of Dyers and Cleaners.

LARSON, C. M., Sinclair Refining Co.

McCULLOUGH, E. W., United States Chamber of Commerce.

POTTER, V. W., Haldeman Co. (Inc.).

PLYLE, ASHBY B.

QUINCY, LYMAN, Sun Oil Co.  
 RADE, F. W., Independent Oil Co.  
 RALPH, HENRY D., Oil, Paint, and Drug Reporter.  
 RUBENSTEIN, S., Rubenstein Co.  
 SCHULTZ, A. G., Addison Cleaning Co. (Inc.).  
 STERN, M. L., Swope's Cleaning Co.  
 TULL, IVAN M., National Association of Dyers and Cleaners.  
 YOUNG, C. F., Youngs Cleaning Works.

#### DEPARTMENT OF COMMERCE:

DE LAGRAVE, D. J., Bureau of Foreign and Domestic Commerce.  
 EMLEY, WARREN E., Bureau of Standards.  
 FAIRCHILD, I. J., Commercial standards unit, Bureau of Standards.  
 HUDSON, R. M., Assistant director, commercial standards group, Bureau of Standards.  
 KENNEDY, DR. H. T., Bureau of Standards.  
 SMITHER, F. W., Bureau of Standards.  
 WALKER, P. H., Bureau of Standards.  
 WATERS, DR. C. E., Bureau of Standards.

### COMMERCIAL STANDARD PROCEDURE

Industry has long sensed the need for a wider application and use of specifications developed and approved by nationally recognized organizations. To assist these bodies and the producers and consumers in securing this result and as a natural outgrowth of the movement toward elimination of waste through simplified practice, the Bureau of Standards has set up a procedure under which specifications, properly indorsed, may be printed as official publications of the Department of Commerce and promulgated as "Commercial Standards." This service parallels that of simplified practice in many respects and is available only upon request.

Broadly speaking, the aim is to continue the same character of cooperative service in this field that is being rendered in simplification. The commercial standards unit is not designed to act as a standardizing body, nor will it engage in the preparation of specifications. Its service is mainly promotional in character, since its chief mission is to get behind a standard or a specification which any industry or its related groups may want to promulgate on a nationwide basis; to determine its eligibility for promulgation; to publish and broadcast it in the event the prerequisites of procedure have been met, including a satisfactory majority acceptance; to facilitate the application of the certification plan for the assurance and convenience of the small purchaser; to provide means for periodical audits of adherence; and to cooperate with the Bureau of Foreign and Domestic Commerce in determining the desire of industry relative to translation and promulgation of such specifications as a basis for foreign commerce.

In general, it may be said that a simplification covers types, sizes, and varieties of a commodity which are retained by industry on the



basis of demand, whereas a commercial standard establishes definite requirements as to grade, quality, or dimensional tolerances in addition to any limitation of variety desired and accepted by the industry.

## ORGANIZATION AND DUTIES OF STANDING COMMITTEE

At the close of its session each general conference appoints a permanent standing committee, composed of not more than three representatives from each phase of the industry; for example, producers, distributors, and consumers.

The proper functioning of the committee requires that its members be able to attend in person meetings held at some central place. They must also be prepared to devote the necessary time and to accept such assignments and responsibilities as may be found requisite to the success of the program.

Because the department in no case assumes the prerogative of taking final action in connection with a commercial standard, it is essential that there be some avenue through which the industry can be consulted promptly and can, in turn, make known its wishes. This function is best performed by a representative standing committee. For this reason the chairman, in accepting his appointment, places his services and those of his committee at the disposal of the Bureau of Standards for the prompt and careful consideration of all questions which may arise when the program is put into actual use.

A committee that quickly and accurately reflects the wishes of the majority of its industry and, through its chairman, promptly disposes of matters referred to it is a practical insurance against any serious difficulty in the adoption of a commercial standard.

The standing committee must recognize that the Department of Commerce has no "police powers" to compel the acceptors to adhere strictly to the letter of the recommendation. Unanimous adoption by the general conference indicates a recognition of the benefits inherent in standardization. If this fact is properly emphasized, the acceptors should be equally willing to follow the program in all cases where it is applicable.

While the recommendation is in effect, the standing committee is to receive all information showing departures, and to apply such corrective measures as appear to be in the best interest of all concerned.

## YOUR COOPERATION

As a producer, distributor, or consumer of some of the commodities which have already been simplified or standardized, you are in a position to add impetus to this method of eliminating waste. The first step in that direction is to record your intention to adhere, as closely as circumstances will allow, to one or more of the existing recommendations *other than those you have already accepted.*

You will, of course, want to examine the schedules before signing. The commercial standards group will, therefore, furnish a copy of any recommendation which you wish to consider with a view to its adoption. Publications no longer available here can be purchased, for a few cents each, from the Superintendent of Documents, Government Printing Office, Washington, D. C. (We will furnish detailed price list on request.)

When you have reached a decision, fill out the acceptance form on page 21, check the proper items on page 22, detach the sheet, and mail it to the commercial standards group, room 316, Department of Commerce, Washington, D. C. In making your selection, remember that commercial standards apply not only to the things you sell but to the things you buy. On the support accorded by the consumer depends, in a large measure, the success of these waste-elimination programs.

The receipt of your signed acceptance will permit the listing of your organization in new editions of the recommendations you have checked. Any proposed revisions will be submitted to you for approval prior to publication.

This support is entirely voluntary and applies to stock items. It is not meant to interfere with the purchase or sale of such special sizes and types as are sometimes required to meet unusual conditions.

Trade associations and individual companies often distribute large numbers of the printed booklets for the information and guidance of their business contacts. In such cases it is possible to extend the scope and degree of adherence by urging each recipient to send in an acceptance. Bear in mind that the practical value of any simplification or standardization is measured by the observance it receives. A number of the simplified practice recommendations have already secured an adherence of more than 90 per cent, by volume, of annual output. If each producer, distributor, and consumer will do his part toward discarding nonessentials and specify simplified lines when buying, adherence will approach 100 per cent. Obviously the higher the adherence to each specific standardization the greater will be the benefits to all concerned.



NOTE.—The information requested in footnotes 1 and 2 is essential to the proper listing of your organization in future editions of the printed bulletins.

## ACCEPTANCE OF COMMERCIAL STANDARDS

Date\_\_\_\_\_

The DEPARTMENT OF COMMERCE,

*Washington, D. C.*

SIRS: We, the undersigned, hereby accept the commercial standards checked on the reverse side hereof <sup>1</sup> as our standards of practice beginning\_\_\_\_\_

(Date)

in the production,<sup>2</sup> distribution,<sup>2</sup> and consumption <sup>2</sup> of the standardized lines.

We will use our best effort to secure their general adoption.

Signed\_\_\_\_\_

Title <sup>3</sup> \_\_\_\_\_

Company <sup>3</sup> \_\_\_\_\_

Street address <sup>3</sup> \_\_\_\_\_

City and State <sup>3</sup> \_\_\_\_\_

<sup>1</sup> Be particular to indicate which commercial standards you are accepting by checking the list on the reverse side of this form.

<sup>2</sup> Please designate by drawing lines through those which do not apply.

<sup>3</sup> Kindly typewrite or print.

### COMMERCIAL STANDARDS

CS. No.	Item
1-28.	Clinical thermometers.
2-29.	Surgical gauze. (In preparation.)
3-28.	Stoddard solvent.
4-29.	Staple porcelain plumbing fixtures. (In preparation.)
5-29.	Steel pipe nipples. (In preparation.)
6-29.	Genuine wrought iron pipe nipples. (In preparation.)
7-29.	Standard weight malleable iron or steel unions. (In preparation.)

(22)







